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IS 998-3 (1983): Methods of chemical analysis of solders (soft and rosin cored) Part 3 Determination of cadmium, zinc, aluminium, bismuth and nickel [MTD 9: Lead, Zinc, Cadmium, Tin, Antimony and their Alloys]

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOLDERS (SOFT AND ROSIN CORED)

PART 3 DETERMINATION OF CADMIUM, ZINC, ALUMINIUM, BISMUTH AND NICKEL

(First Revision)

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Indian Standard

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PART 3 DETERMINATION OF CADMIUM, ZINC, ALUMINIUM, BISMUTH AND NICKEL

(First Revision)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOLDERS (SOFT AND ROSIN CORED)

PART 3 DETERMINATION OF CADMIUM, ZINC, ALUMINIUM, BISMUTH AND NICKEL

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 3) (First Revision) was adopted by the Indian Standards Institution on 26 April 1983, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 21 grades of soft solders and 5 grades of rosin cored solders have been specified in IS : 193-1982* and IS : 1921-1975† respectively. In order to determine correctly the composition of different solders specified in these standards, methods of analysis have been described in various parts of this standard. Due consideration has also been given in preparation of this standard to the facilities available in the country for such analysis.

0.2.1 This standard was first published in 1959. In this revision, the standard has been divided in four parts. Methods for determination of bismuth and silver have been included in this part and Part 4 of this standard respectively. In this revision, preparation of sample for the analysis of rosin cored solders covered in IS : 1921-1975†, has also been included. This part covers the determination of cadmium, zinc, aluminium, bismuth and nickel. The other parts cover the following:

Part 1	Determination of tin, antimony and lead;
Part 2	Determination of iron, copper and arsenic; and
Part 4	Determination of lead and silver.

0.2.2 Method for determination of cadmium stipulated in earlier edition has been substituted by photometric method.

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*Specification for soft solder (*fourth revision*).

†Specification for rosin cored solder wire (*first revision*).

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 3) covers photometric methods of determination of cadmium, zinc, aluminium, bismuth and nickel in lead and tin base solders (*see* IS : 193-1982† and IS : 1921-1975‡) having chemical composition within the following range:

Constituents	*Percent*
Cadmium	0·005 *Max*
Zinc	0·003 *Max*
Aluminium	0·001 *Max*
Bismuth	0 05 *Max*
Nickel	0·002 *Max*

2. SAMPLING FOR CHEMICAL ANALYSIS

2.1 Samples shall be drawn in accordance with the procedure laid down in IS : 193-1982† or IS : 1921-1975‡.

2.2 Preparation of Sample (for Rosin Cored Solder) — Samples shall be prepared as mentioned below.

2.2.1 From each reel or coil taken in accordance with **2.1**, cut a length of solder of mass approximately 50 g.

2.2.2 Melt the five lengths in a clean silica crucible at a temperature just above the liquidus temperature of the alloy and mix thoroughly, using a stirrer that will not contaminate the melt. After the flux has floated to the top, carefully pour the alloy into a cool mould so as to form a bar approximately 6 mm thick that does not contain any flux. Clean the bar by washing with methanol to remove the flux residue and then wipe it dry thoroughly and then thoroughly wiping dry.

2.2.3 Make cuts with a clean, fine saw across the entire width of the bar at regular intervals along its length. Do not use any lubricant during this operation. Treat the sawings carefully with a magnet to remove any particles of steel that may have been introduced during sawing. Mix the sawings thoroughly.

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*Rules for rounding off numerical values (*revised*).

†Specification for soft solder (*fourth revision*).

‡Specification for rosin cored solder wire (*first revision*).

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water, conforming to IS : 1070-1977*, shall be used, where the use of water as a reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the result of analysis.

4. DETERMINATION OF CADMIUM

4.1 Outline of the Method — After removal of all interfering elements, dithizone is added to form a coloured complex with cadmium which is measured photometrically at a wavelength of 510 nm.

4.2 Apparatus — Any instrument suitable for measuring the absorbance of the solution at a wavelength of 510 nm may be used together with 4 cm cell.

4.3 Reagents

4.3.1 *Bromine-Hydrobromic Acid Mixture* — To 90 ml of hydrobromic acid add 5 ml of bromine, shake and allow to stand until saturated.

4.3.2 *Perchloric Acid* — rd = 1·54.

4.3.3 *Sodium Tartrate Solution* — 20 percent (*m/v*). Dissolve 20 g of sodium tartrate in water and dilute to 100 ml.

4.3.4 *Methyl Red Indicator Solution* — 0·02 percent (*m/v*). Dissolve 0·02 g of methyl red in water and dilute to 100 ml.

4.3.5 *Sodium Hydroxide Solution* — 20 percent (*m/v*). Dissolve 20 g of sodium hydroxide in water and dilute to 100 ml.

4.3.6 *Acetic Acid* — glacial.

4.3.7 *Cupferron Solution* — 2 percent (*m/v*). Dissolve 2 g of cupferron in water and dilute to 100 ml.

4.3.8 *Chloroform*

4.3.9 *Bromine Water* (*Saturated*) — To 100 ml of water, add 5 ml of bromine, shake and allow to stand until saturated.

4.3.10 *Hydroxyammonium Chloride Solution* — 10 percent (*m/v*). Dissolve 10 g of hydroxyammonium chloride in water and dilute to 100 ml.

4.3.11 *Potassium Iodide Solution* — 10 percent (*m/v*). Dissolve 10 g of potassium iodide in water and dilute to 100 ml.

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*Specification for water for general laboratory use (*second revision*).

4.3.12 *Brucine Sulphate Solution* — 2 percent (*m/v*). Dissolve 2 g of brucine sulphate by warming in 100 ml of acetic acid (5 percent *v/v*).

4.3.13 *Dilute Hydrochloric Acid* — 5 percent (*v/v*). Dilute 5 ml of concentrated hydrochloric acid (rd = 1·16 to 1·18) to 100 ml.

4.3.14 *Dithizone Solution* — Dissolve 25 mg of dithizone in 25 ml of chloroform. Shake twice with 100 ml of ammonia solution (2 percent *v/v*) (*see* Note) in a large separating funnel and discard the chloroform layer. Make the aqueous solution just acidic by addition of concentrated hydrochloric acid (rd = 1·16). Add 250 ml of carbon tetrachloride, shake, and separate the carbon tetrachloride layer. Repeat this step. Combine the two carbon tetrachloride extracts, wash with water and store in a dark bottle.

NOTE — Preferably, ammonia gas should be dissolved in demineralized water.

4.3.15 *Carbon Tetrachloride*

4.3.16 *Dilute Hydrochloric Acid* — 50 percent (*v/v*). Dilute 50 ml of concentrated hydrochloric acid (rd = 1·16) to 100 ml.

4.3.17 *Standard Cadmium Solution (1 ml = 0·5 mg of Cadmium)* — Dissolve 0·250 g of cadmium in 100 ml of nitric acid (20 percent *v/v*), transfer to 500-ml volumetric flask and dilute to the mark.

4.3.18 *Standard Cadmium Solution (1 ml = 0·005 mg of Cadmium)* — Transfer 5 ml of standard cadmium solution (*see* **4.3.17**) to a 500-ml volumetric flask and dilute to the mark.

4.4 Procedure

4.4.1 Weigh 1·25 g of sample and transfer to a 150-ml beaker. Add 20 ml of the bromine-hydrobromic acid mixture and 5 ml of perchloric acid. Evaporate to fuming, and if the solution is cloudy add a further quantity of the bromine-hydrobromic acid mixture. Repeat until the solution is clear, indicating complete removal of tin and antimony. Cool, dilute, transfer to a 250-ml volumetric flask and dilute to the mark. Transfer a 5 ml aliquot to a 100-ml separating funnel, add 2 ml of sodium tartrate solution, one drop of methyl red indicator solution and make slightly alkaline with sodium hydroxide solution and subsequently acidify with 2 ml of acetic acid (glacial). Dilute to 30 ml, add 5 ml of cupferron solution and shake with 10 ml of chloroform. Allow to separate and discard the chloroform layer. Repeat the addition of cupferron solution and extraction with chloroform until no precipitate appears on the addition of the cupferron solution. To the aqueous solution remaining in the funnel add 1 ml of bromine water and, after one minute extract the excess of bromine by shaking with two 5 ml portions of chloroform. Add 1 ml of hydroxyammonium chloride solution. Then add 5 ml of potassium iodide solution and 5 ml of brucine sulphate solution. Extract three times with 15 ml portions of chloroform and collect the chloroform extracts in a 200 ml separating funnel. Discard the aqueous layer in the first funnel. Shake the extracts in the second funnel with 20 ml of dilute hydrochloric acid (5 percent). Run off the chloroform layer into another funnel and shake with a further 10 ml of hydrochloric acid (5 percent). Discard the chloroform layer and combine the two hydrochloric acid extracts.

4.4.2 Add 30 ml of sodium hydroxide solution and 20 ml of the dithizone solution and shake for 1 minute. Remove any droplets of water from the stem of the funnel by inserting a roll of filter paper. Remove the paper and run off the carbon tetrachloride layer into a small stoppered bottle.

4.4.3 Measure the absorbance of the solution at 20 ± 1°C using the conditions specified in **4.2** and using carbon tetrachloride in the reference cell.

4.4.4 Carry out simultaneously, absorbance determination for corresponding reagent blank.

4.4.5 Deduct the blank reading and calculate the percentage of cadmium in the sample by reference to the appropriate calibration curve (*see* **4.5**).

4.5 Calibration Curve

4.5.1 Transfer to a series of 100-ml separating funnels 0·5, 1·0, 1·5, and 2·0 ml of standard cadmium solution (1 ml = 0·005 mg of cadmium).

4.5.2 Dilute to 30 ml and proceed as given in **4.4.2** to **4.4.4**. Prepare the calibration curve by plotting absorbances against the mass of cadmium in the various aliquots after deducting the blank reading.

4.6 Calculation — Calculate the percentage of cadmium as follows:

$$\text{Cadmium, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of cadmium found in the sample solution, as determined from the calibration curve; and

B = mass in g of the sample used.

NOTE — In presence of more than 10 percent of lead, remove the bulk of lead as follows:

Cool, dilute with 20 ml of water, add 5 ml of dilute hydrochloric acid (50 percent) and cool in ice-water. Filter off the lead chloride on to a sintered glass filter (porosity 4) and wash with a little cold dilute hydrochloric acid (10 percent). Evaporate the filtrate to fumes of perchloric acid and continue as in the procedure.

5. DETERMINATION OF ZINC

5.1 Outline of the Method — After removal of all interfering elements, dithizone is added to form a coloured complex with zinc which is measured photometrically at 535 nm.

5.2 Apparatus — Any instrument suitable for measuring the absorbance of the solution at a wavelength of 535 nm may be used together with 4 cm cell.

5.3 Reagents

5.3.1 *Bromine-Acid Mixture* — To 45 ml of hydrochloric acid (rd = 1·16 to 1·18) and 45 ml of hydrobromic acid (rd = 1·46 to 1·49), add carefully 10 ml of bromine.

5.3.2 *Perchloric Acid* — rd = 1·54.

5.3.3 *Dilute Hydrochloric Acid* — 50 percent (*v/v*). Dilute 50 ml of concentrated hydrochloric acid (rd = 1·16) to 100 ml.

5.3.4 *Dilute Hydrochloric Acid* — 25 percent (*v/v*). Dilute 25 ml of concentrated hydrochloric acid (rd = 1·16) to 100 ml.

5.3.5 *Dilute Hydrochloric Acid* — 10 percent (*v/v*). Dilute 10 ml of concentrated hydrochloric acid (rd = 1·16) to 100 ml.

5.3.6 *Hydrogen Sulphide* — gas.

5.3.7 *Dilute Nitric Acid* — 50 percent (*v/v*). Dilute 50 ml of concentrated nitric acid (rd = 1·42) to 100 ml.

5.3.8 *Methyl Red Indicator Solution* — 0·05 percent (*m/v*). Dissolve 0·05 g of water soluble methyl red in 100 ml of water.

5.3.9 *Sodium Tartrate Solution* — 20 percent (*m/v*). Dissolve 20 g of sodium tartrate in 70 ml of water and dilute to 100 ml.

5.3.10 *Ammonia Solution* — 50 percent (*v/v*). Dilute 50 ml of ammonia solution (rd = 0·91) to 100 ml.

5.3.11 *Sodium Cyanide Solution* — 5 percent (*v/v*). Dissolve 5 g of sodium cyanide in 50 ml of water and dilute to 100 ml.

5.3.12 *Acetic Acid* — 50 percent (*v/v*). Dilute 50 ml of glacial acetic acid to 100 ml.

5.3.13 *Bis (2-Hydroxyethyl) dithicarbamate* — Dissolve 4·0 g of diethanolamine and 1·0 ml of carbon disulphide in 40 ml of methanol. Keep the solution for 3 to 4 days only.

5.3.14 *Dithizone Solution* — 0·005 percent (*m/v*). Dissolve 0·025 g of dithizone in 25 ml of chloroform and shake with 100 ml of ammonia solution (2 percent) for 1 minute in a separating funnel. Separate the two layers and shake the chloroform layer with a further 100 ml of ammonia solution (2 percent) for one minute. Discard the chloroform layer, combine the two aqueous layers and wash with 20 ml of carbon tetrachloride. Make the aqueous solution just acidic with dilute hydrochloric acid (50 percent) and shake with 250 ml of carbon tetrachloride. Separate the carbon tetrachloride layer, add a further 250 ml of carbon tetrachloride and shake again. Separate and combine the carbon tetrachloride extracts, wash them with water ~~and~~ store in a dark bottle.

5.3.15 *Sodium Sulphide Solution* — 0·04 percent (*m/v*). Dissolve 0·4 g of sodium sulphide in 50 ml of freshly boiled distilled water and dilute to 1 litre with freshly boiled distilled water. Prepare fresh solution each day.

5.3.16 *Ammonia Solution* — 2 percent (*v/v*). Dilute 2 ml of ammonia solution (rd = 0·91) to 100 ml.

5.3.17 *Standard Zinc Solution (1 ml = 0·1 mg of Zinc)* — Dissolve 0·100 g of zinc in 50 ml of hydrochloric acid (25 percent), transfer to a 1-litre volumetric flask and dilute up to the mark.

5.3.18 *Standard Zinc Solution (1 ml = 0·005 mg of Zinc)* — Transfer 25·0 ml of standard zinc solution (*see* **5.3.17**) to a 500-ml volumetric flask and dilute up to the mark.

5.3.19 *Standard Zinc Solution (1 ml = 0·001 mg of Zinc)* — Transfer 20·0 ml of standard zinc solution (*see* **5.3.18**) to a 100-ml volumetric flask and dilute up to the mark.

5.4 Procedure

5.4.1 Weigh accurately 1 g of the sample and transfer to a 150-ml beaker. Add 20 ml of the bromine-acid mixture and 5 ml of perchloric acid (rd = 1·54). Evaporate to fuming and if the solution is cloudy add a further 5 ml of the bromine-acid mixture. Repeat the evaporation until the solution is clear.

5.4.2 Cool, add 20 ml of water and 5 ml of dilute hydrochloric acid (50 percent) and cool again in ice. Filter off any lead chloride through a sintered glass filter (porosity 4) and wash with chilled dilute hydrochloric acid (10 percent). Transfer the filtrate to a 100-ml volumetric flask and dilute to the mark.

5.4.3 Transfer a 25 ml aliquot to a 100-ml beaker and pass hydrogen sulphide gas for 10 to 15 minutes. Filter through a medium texture filter paper and wash the precipitate two or three times with hot water. Evaporate the filtrate to less than 20 ml, oxidize with two drops of dilute nitric acid, cool and transfer to a 100-ml separating funnel.

5.4.4 Dilute to about 25 ml, add 1 drop of methyl red indicator solution, 2 ml of sodium tartrate solution then ammonia solution (50 percent), dropwise until the colour changes to yellow. Add 1 ml of sodium cyanide solution, then acetic acid, dropwise until the neutral peach colour is obtained. Extract the methyl red by shaking the solution with 5 ml of carbon tetrachloride, allow the layers to separate and discard the carbon tetrachloride layer.

5.4.5 Add 1 ml of the bis (2 hydroxyethyl) dithicarbamate solution and extract the zinc with dithizone solution.

5.4.6 Shake with 10 ml of dithizone solution for 1 minute. Allow the layers to separate and run the lower carbon tetrachloride layer into a second separating funnel. Shake the aqueous layer with 5 ml portions of dithizone solution, running each successive extract into the second separating funnel until there is no change in the colour of the dithizone solution. To the aqueous layer remaining in the first funnel, add 5 ml of carbon tetrachloride, shake, allow to separate and add the carbon tetrachloride layer to the combined extracts. Discard the aqueous layer.

5.4.7 Shake the combined dithizone extracts (combined carbon tetrachloride layers) with 20 ml of sodium sulphide solution for 30 seconds, allow the layers to separate and discard the aqueous layer. Repeat this extraction with further 10 ml portions of sodium sulphide solution until the upper aqueous layer is colourless. Remove any droplets of water from the stem of the separating funnel by inserting a roll of filter paper. Remove the paper and run off the carbon tetrachloride layer into a dry 50-ml volumetric flask. Wash the aqueous layer with carbon tetrachloride, allow the layers to separate, then transfer the carbon tetrachloride layer to the same volumetric flask. Make up to the mark with carbon tetrachloride.

5.4.8 Measure the absorbance of the solution using the conditions specified in **5.2**.

5.4.9 Carry out simultaneously, absorbance determination for corresponding reagent blank.

5.4.10 Deduct the blank reading and calculate the percentage of zinc in the sample by reference to the calibration curve (*see* **5.5**).

5.5 Calibration Curve

5.5.1 Transfer to a series of separating funnels 2·0, 4·0, 6·0, 8·0 and 10 ml of standard zinc solution (1 ml = 0·001 mg of zinc), for 4 cm cell.

5.5.2 Proceed as given in **5.4.4** to **5.4.9**. Prepare calibration curve by plotting absorbance against the quantity of zinc in the various aliquots, after deduction of the blank reading.

5.6 Calculation — Calculate the percentage of zinc as follows:

$$\text{Zinc, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of zinc in the sample solution, as determined from the calibration curve; and

B = mass in g of the sample used.

6. DETERMINATION OF ALUMINIUM

6.1 Outline of the Method — After removal of all interfering elements, chrome azurol-S is added to form a coloured complex with aluminium which is measured photometrically at 550 nm.

6.2 Apparatus — Any instrument suitable for measuring the absorbance of the solution at a wavelength of 550 nm may be used together with 1 cm cell.

6.3 Reagents

6.3.1 *Bromine-Acid Mixture* — To 45 ml of hydrochloric acid (rd = 1·16) and 45 ml of hydrobromic acid (rd = 1·46 to 1·49), add carefully 10 ml of bromine.

6.3.2 *Dilute Nitric Acid* — 50 percent (*v/v*). Dilute 50 ml of concentrated nitric acid (rd = 1·42) to 100 ml.

6.3.3 *Dilute Sulphuric Acid* — 50 percent (*v/v*). To 40 ml of water add cautiously 50 ml of concentrated sulphuric acid (rd = 1·84) mix, cool and dilute to 100 ml.

6.3.4 *Dilute Sulphuric Acid* — 2 percent (*v/v*). To 40 ml of water add cautiously 2 ml of concentrated sulphuric acid (rd = 1·84) mix, cool and dilute to 100 ml.

6.3.5 *Hydrogen Sulphide* — gas.

6.3.6 *Hydrogen Sulphide-Sulphuric Acid* — Pass hydrogen sulphide through dilute sulphuric acid (2 percent) for 10 minutes.

6.3.7 *Dilute Hydrochloric Acid* — 10 percent (*v/v*). Dilute 10 ml of concentrated hydrochloric acid (rd ≏ 1·16) to 100 ml.

6.3.8 *Sodium Hydroxide Solution* — 5 percent (*m/v*). Dissolve 5 g of sodium hydroxide in water and dilute to 100 ml.

6.3.9 *Ascorbic Acid Solution* — 5 percent (*m/v*). Dissolve 5 g of ascorbic acid in water and dilute to 100 ml. This solution should be prepared freshly each day.

6.3.10 *Chrome Azurol-S* — 0·3 percent (*m/v*). Dissolve 0·3 g of chrome-azurol-S in water and dilute to 100 ml. The solution should be prepared freshly each day.

6.3.11 *Ammonia Solution* — 10 percent (*v/v*). Dilute 10 ml of ammonia solution (rd = 0·91) to 100 ml.

6.3.12 *Buffer Solution* — To 850 ml of sodium acetate solution (*see* **6.3.12.1**) add 150 ml of acetic acid (*see* **6.3.12.2**) and mix.

6.3.12.1 *Sodium acetate solution* — 2·72 percent (*v/v*). Dissolve 27·2 g of sodium acetate ($CH_3COONa \cdot 3H_2O$) in water and dilute to 1 litre.

6.3.12.2 *Acetic acid solution* — 1·16 percent (*v/v*). Dilute 11·6 ml of glacial acetic acid to 1 litre.

6.3.13 *Standard Aluminium Solution (1 ml = 0·1 mg of Aluminium)* — Dissolve 1·758 9 g of aluminium potassium sulphate [$Al_2(SO_4)_3 K_2SO_4 \cdot 24 H_2O$] in water, transfer to a 1-litre volumetric flask and dilute up to the mark.

6.3.14 *Standard Aluminium Solution (1 ml = 0·005 mg of Aluminium)* — Transfer 25 ml of standard aluminium solution (*see* **6.3.13**) to a 500-ml volumetric flask and dilute up to the mark.

6.3.15 *Bromine-Hydrochloric Acid* — To 90 ml of concentrated hydrochloric acid (rd = 1·16) add carefully 10 ml of bromine.

6.4 Procedure

6.4.1 Weigh accurately 1 g of sample and transfer to a 150-ml beaker. Add 10 ml of the bromine-acid mixture and evaporate it to dryness.

6.4.2 Dissolve the residue in 5 ml of dilute nitric acid (50 percent), cool, add 2 ml of dilute sulphuric acid (50 percent) and evaporate to fuming. Cool, dilute to 20 ml, simmer for 5 minutes and allow the precipitate to settle for 1 hour. Filter through a sintered glass filter (porosity 4) washing with small quantities of dilute sulphuric acid (2 percent). Pass hydrogen sulphide gas through the warm filtrate for 15 minutes. Filter off any precipitate and wash with small quantities of hydrogen sulphide-sulphuric acid, collecting the filtrate in a 100-ml beaker.

6.4.3 Evaporate to fuming and continue fuming until free from sulphuric acid. Dissolve the residue in 4 ml of dilute hydrochloric acid (10 percent) and add 20 ml of water. Add sodium hydroxide solution (5 percent) until the *p*H is approximately 2·5. This should require about 3 to 4 ml of sodium hydroxide solution. Transfer to a 50-ml volumetric flask and dilute up to the mark.

6.4.4 Transfer a suitable aliquot to a 50-ml volumetric flask, add 1 ml of ascorbic acid solution, 0·5 ml of chrome-azurol-S solution, dilute to 20 ml and add ammonia solution until the colour changes to yellow. Add 20 ml of buffer solution, dilute up to the mark and allow to stand for 5 to 10 minutes.

6.4.5 Measure the absorbance of the solution at 20 ± 1°C using the conditions specified in **6.2**.

6.4.6 Carry out simultaneously absorbance determination for corresponding reagent blank.

6.4.7 Deduct the blank reading and calculate the percentage of aluminium in the sample by reference to the calibration curve (*see* **6.5**).

6.5 Calibration Curve

6.5.1 Transfer to a series of 100-ml beakers 1·0, 2·0, 3·0 and 4·0 ml of standard aluminium solution (1 ml = 0·005 mg of aluminium), and continue as given in **6.4.3** to **6.4.6**.

6.5.2 Prepare a calibration curve by plotting absorbance against the mass of aluminium in the various aliquots after deduction of the blank reading.

6.6 Calculation — Calculate the percentage of aluminium as follows:

$$\text{Aluminium, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of aluminium in sample solution, as determined from the calibration curve; and

B = mass in g of the sample used.

NOTE — In the presence of more than 15 percent of lead remove the bulk of the lead as follows:

Dissolve the sample in 20 ml of the bromine-hydrochloric acid, evaporate to about 3 ml, dilute with 20 ml of water and cool in ice. Filter off the lead chloride on to a sintered glass filter (porosity 4) and wash with small quantities of ice-cold hydrochloric acid. Evaporate the filtrate to about 5 ml, add 10 ml of the bromine-acid mixture and repeat the evaporation to dryness. Continue from **6.4.2** of the procedure.

7. DETERMINATION OF BISMUTH

7.1 Outline of the Method — After removal of interfering elements, thiourea solution is added to form a yellow coloured complex with bismuth which is measured photometrically at 440 nm.

7.2 Apparatus — Any instrument suitable for measuring the absorbance of the solution at a wavelength of 440 nm may be used with 1 cm cell.

7.3 Reagents

7.3.1 *Bromine-Hydrobromic Acid Mixture* — Pour 20 ml of bromine into 180 ml of hydrobromic acid.

7.3.2 *Perchloric Acid-Phosphoric Acid Mixture* — Mix 840 ml of perchloric acid (70 percent) with 160 ml of phosphoric acid.

7.3.3 *Fluoboric Acid Solution* — Pour 5 ml of hydrofluoric acid into 45 ml of a standard solution of boric acid and dilute to 50 ml.

7.3.4 *Thiourea Solution (80 g/l)* — Dissolve 16·00 g of thiourea in 175 ml of cold water. Filter and wash into a 200-ml volumetric flask, dilute up to the mark and mix. This solution shall be freshly prepared.

7.3.5 *Standard Bismuth Solution (1 ml = 0·1 mg Bismuth)* — Transfer 0·100 g of bismuth metal to a 150-ml beaker and add 10 ml of nitric acid (1:3). Heat gently to dissolve the sample and to expel brown fumes. Cool and dilute with nitric acid (1:19) to one litre in a volumetric flask.

7.4 Procedure

7.4.1 Transfer 2 g of the sample to a 250-ml wide mouth flask and add 20 ml of bromine-hydrobromic acid mixture, cover and heat gently to dissolve the sample completely. When dissolution is complete, add 12 ml of perchloric acid-phosphoric acid mixture and heat carefully over an open flame to expel hydrobromic acid. Convert the salts to perchlorates. If the solution is cloudy add 10 ml of hydrobromic acid and heat gently. Finally, when a fairly clear solution is obtained, expel all hydrobromic acid. Add a few drops of nitric acid and fume till the volume is reduced to about 2 ml. Cool to room temperature, add a mixture of 40 ml of water and 5 ml of fluoboric acid solution, mix and add quickly from a pipette 25 ml of freshly prepared thiourea solution. Transfer the solution to a 100-ml volumetric flask, cool to room temperature, make up to the mark with water and mix.

7.4.2 Measure the absorbance of the solution against the corresponding reagent blank at a wavelength of 440 nm.

7.5 Calibration Curve — Take a series of standard bismuth solutions and proceed as described under **7.4.1** and **7.4.2**. Measure the absorbance of all the standard solutions against the corresponding reagent blank. Construct the calibration curve by plotting the corrected photometric reading against the quantities of bismuth in the range of 0·1 mg to 2·0 mg (in 100 ml final volume).

7.6 Calculation — Calculate the percentage of bismuth as follows:

$$\text{Bismuth, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of bismuth in the sample solution, as determined from the calibration curve; and

B = mass in g of the sample used.

NOTE — Lead, copper and certain other heavy metals form soluble complexes with thiourea solution. It is essential, in the present method, to use 440 nm filter with a sufficiently narrow band width, otherwise higher result may be obtained.

8. DETERMINATION OF NICKEL BY THE DIMETHYLGLYOXIME METHOD

8.1 Outline of the Method — After removal of all interfering elements by suitable separations, nickel is determined photometrically by measuring, at approximately 530 nm, the red coloured soluble complex formed with dimethylglyoxime.

8.2 Concentration Range

8.2.1 The recommended concentration range is from 0·02 to 0·5 mg of nickel in 100 ml of solution, using a cell depth of 2 cm.

8.3 Reagents

8.3.1 *Perchloric Acid* — 70 percent.

8.3.2 *Standard Nickel Solution (1 ml = 0·02 mg Nickel)* — Dissolve 0·2 g of nickel (purity 99·9 percent, *Min*) in 20 ml of dilute nitric acid (1:1) with the aid of gentle heat. Cool, transfer to a litre volumetric flask, make up to the mark with water, and mix. Dilute 100 ml of this solution to 1 litre in volumetric flask with water and mix.

8.3.3 *Citric Acid Solution* — 300 g/l.

8.3.4 *Bromine Water* — saturated.

8.3.5 *Dilute Ammonium Hydroxide Solution* — 1:1 (*v/v*).

8.3.6 *Alcohol Solution of Dimethylglyoxime (10 g/l)* — Dissolve 10 g of dimethylglyoxime in ethanol and dilute to 1 litre.

8.3.7 *Test-Lead* — Lead containing under 0·001 percent of nickel.

8.3.8 *Bromine-Hydrobromic Acid Mixture* — Pour 20 ml of bromine into 180 ml of hydrobromic acid.

8.4 Procedure

8.4.1 Depending on the nickel content, transfer up to 2·0 g of the accurately weighed sample to a suitable vessel. Add up to 20 ml of bromine-hydrobromic acid mixture. Cover and heat gently until dissolution of the sample is complete avoiding excessive loss of bromine. If necessary, add a few extra drops of bromine to ensure complete dissolution of the sample. Add 10 ml of perchloric acid and heat with continuous swirling over an open flame in a well ventilated hood to expel tin and antimony bromides. When copious white fumes of perchloric acid appear, add a few drops of nitric acid, heat gently and intermittently to decompose any perchloric acid and reduce the volume to about 2 ml.

8.4.2 Add 40 ml of water, 1 g of test lead to the flask and boil for 16 minutes. Filter through a fine filter paper to remove lead, antimony and copper. Wash with water and discard the precipitate. Transfer the solution to a 100-ml volumetric flask.

8.4.3 Dilute to 50 ml and add, successively (swirling the flasks between additions) 10 ml of citric acid solution, 5 ml of bromine water, and just sufficient ammonium hydroxide solution to bleach the bromine colour. Add 3 ml of ammonium hydroxide solution in excess and cool to room temperature. Add 3 ml of dimethylglyoxime solution, dilute to the mark and mix. The addition of bromine water, ammonium hydroxide solution and dimethylglyoxime should be made without interruption and the subsequent photometric measurement should be made within 10 ± 2 minutes after addition of the dimethylglyoxime.

8.4.4 Measure the absorbance of the solution against the corresponding reagent blank at a wavelength of 530 nm.

8.5 Calibration Curve

8.5.1 Transfer 1·0, 2·0, 4·0, 6·0, 10·0, 15·0, 20·0 and 25·0 ml of nickel solution (1 ml = 0·02 mg Ni) to eight 100-ml volumetric flasks. Add 2 ml of perchloric acid to each flask. Proceed in accordance with **8.4.3** and **8.4.4**.

8.5.2 Prepare the calibration curve by plotting absorbance against the mass of nickel in the various aliquots after deducting the blank reading.

8.5.3 *Calculation* — Calculate the percentage of nickel as follows:

$$\text{Nickel, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mg of nickel found in the sample solution, as determined by the calibration curve; and

B = g of sample represented by the sample or aliquot used.

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